checked by TT on 1/13/15

MEMORANDUM

TO: Mr. Addison Rice

Anderson, Mulholland and Associates

DATE: January 7, 2015

FROM: R. Infante

FILE: 1412151B

RE:

Data Validation
Air samples
SDG: 1412151B

SUMMARY

Full validation was performed on the data for several gas samples analyzed for Methane by method ASTM Method D-1946: Standard Practice for Analysis of Reformed Gas by Gas Chromatography. The samples were collected at the Bristol Myer Squib-Building 5 VI facility, Humacao, PR site on December 08-09, 2014 and submitted to Eurofins Air Toxics, Inc. of Folson, California that analyzed and reported the results under delivery group (SDG) 1412151B.

The sample results were assessed according to USEPA data validation guidance documents in the following order of precedence: ASTM Method D-1946: Standard Practice for Analysis of Reformed Gas by Gas Chromatography; Validating Air Samples. Volatile Organic Analysis of Ambient Air in Canisters by Method TO-15, (SOP # HW-31. Revision #4. October, 2006 The QC criteria and data validation actions listed on the data review worksheets are from the primary guidance document, unless otherwise noted.

In general the data is valid as reported and may be used for decision making purposes. The data results are acceptable for use.

SAMPLES

The samples included in the review are listed below

Client Sample ID	Lab. Sample ID	Collected Date	Matrix	Analysis
BSIA-5 (2014) BSIA-3D (2014) BSIA-3 (2014) BSIA-11 (2014) BSIA-9 (2014)	======================================	12/08/2014 12/09/2014 12/09/2014 12/09/2014 12/09/2014	Air Air Air Air Air Air Air Air	Methane Methane Methane Methane Methane Methane Methane

REVIEW ELEMENTS

Sample data were reviewed for the following parameters, where applicable to the method

- o Agreement of analysis conducted with chain of custody (COC) form
- o Holding time and sample preservation
- o Initial and continuing calibrations
- Method blanks/trip blanks/field blank
- o Canister cleaning certification criteria
- o Field duplicate results
- Laboratory control sample/laboratory control sample duplicate (LCS/LCSD) results
- o Quantitation limits and sample results

DISCUSSION

Agreement of Analysis Conducted with COC Request

Sample reports corresponded to the analytical request designated on the chain-of-custody form.

Holding Times and Sample Preservation

Sample preservation was acceptable.

Samples analyzed within method recommended holding time.

Initial and Continuing Calibrations

Methane (ASTM Method D-1946)

The percent relative standard deviations (%RSDs) for response factors (RFs) of all target analytes were within the QC acceptance criteria in the initial calibration. Ongoing accuracy of the instrument was determined by the analysis of a continuing calibration standard.

Method Blank/Trip Blank/Field Blank

Target analytes were not detected in laboratory method blanks for VOCs.

Summa canister met cleaning certification criteria.

Laboratory/Field Duplicate Results

VOCs

Field duplicate were analyzed as part of this data set. RPD was outside the 25 % method criteria; RPD for field duplicate samples was 36%. Results for sample and sample duplicate qualified as estimated (j).

LCS/LCSD Results

VOCs

LCS/LCSD (blank spikes) were analyzed by the laboratory associated with this data package. Recoveries and RPD were within laboratory control limits.

Quantitation Limits and Sample Results

Dilutions were not required with this data set.

Méndez

IC # 1888

Calculations were spot checked.

Certification

Rafael Infante

Chemist License 18

The following samples 1412151B-01A; 1412151B-02A; 1412151B-03A; 1412151B-04A; and 1412151B-05A were analyzed following standard procedures accepted by regulatory agencies. The quality control requirements met the methods criteria except in the occasions described in this document. The results are valid.

1507624



Client Sample ID: BSIA-5 (2014) Lab ID#: 1412151B-01A

NATURAL GAS ANALYSIS BY MODIFIED ASTM D-1946

File Name: Dil. Factor:	9121514 1.58		ition: 12/8/14 3:55:00 PM sis: 12/15/14 05:50 PM
Dii. Factor.	1.30	Rpt. Limit	Amount
Compound		(%)	(%)
Methane		0.00016	0.0019





Client Sample ID: BSIA-3D (2014) Lab ID#: 1412151B-02A

NATURAL GAS ANALYSIS BY MODIFIED ASTM D-1946

File Name: Dil. Factor:	9121515 2.87		ition: 12/9/14 9:30:00 AN sis: 12/15/14 06:11 PM
		Rpt. Limit	Amount
Compound		(%)	(%)
Methane		0.00029	0.0012





Client Sample ID: BSIA-3 (2014) Lab ID#: 1412151B-03A

NATURAL GAS ANALYSIS BY MODIFIED ASTM D-1946

File Name:	9121516	Date of Collec	tion: 12/9/14 9:20:00 AN
Dil. Factor:	1.49		sis: 12/15/14 06:57 PM
		Rpt. Limit	Amount
Compound		(%)	(%)
Methane		0.00015	0.0014





Client Sample ID: BSIA-11 (2014) Lab ID#: 1412151B-04A

NATURAL GAS ANALYSIS BY MODIFIED ASTM D-1946

File Name: Dil. Factor:	9121517 1.83		ction: 12/9/14 10:46:00 AN
Compound		Rpt. Limit (%)	Amount (%)
Methane		0.00018	0.00031





Methane

Air Toxics

Client Sample ID: BSIA-9 (2014) Lab ID#: 1412151B-05A

NATURAL GAS ANALYSIS BY MODIFIED ASTM D-1946

 File Name:
 9121518
 Date of Collection:
 12/9/14 10:50:00 AM

 Dil. Factor:
 1.61
 Date of Analysis:
 12/15/14 07:41 PM

 Rpt. Limit
 Amount

 Compound
 (%)
 (%)

0.00016

Container Type: 6 Liter Summa Canister (100% Certified)



0.00032

Project Manager Terry

Collected by: (Print and Sign)

Sample Transportation Notice
Relinquishing signature on this document indicates that sample is being shipped in compilance witt all applicable local, State, Federal, national, and international laws, regulations and ordinances cany kind. Air Toxics Limited assumes no liability with respect to the collection, handling or shippin of these samples. Relinquishing signature also indicates agreement to hold harmless, defended to the collection of any kind, related to the and indemnify Air Toxics Limited against any claim, demand, or action, of any kind, related to the collection, handling, or shipping of samples. D.O.T. Hotline (800) 467-4922

Project Info:

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	Project Number:1412151B
	Date:12/08-09/2014
REVIEW OF VOLATILE ORO The following guidelines for evaluating volatile organics actions. This document will assist the reviewer in using guidecision and in better serving the needs of the data users. USEPA data validation guidance documents in the following D-1946 method for measuring permanent gases and light samples using gas chromatography (GC) and a thermal condetection (FID). Validating Air Samples. Volatile Organic ATO-15, (SOP # HW-31. Revision #4. October, 2006). The other data review worksheets are from the primary guidance do The hardcopied (laboratory name) _Eurofinsreviewed and the quality control and performance data summer reviewed.	were created to delineate required validation professional judgment to make more informed. The sample results were assessed according to g order of precedence: QC criteria from ASTM at hydrocarbons in refinery and other sources aductivity detector (TCD) and/or flame ionization. Analysis of Ambient Air in Canisters by Method QC criteria and data validation actions listed or occument, unless otherwise noted. data package received has been
Lab. Project/SDG No.:1412151B	
No. of Samples:5	-
Trip blank No.: Field blank No.: Equipment blank No.: Field duplicate No.:1412151B-02A/1412151B-03A	
X Data CompletenessX Holding TimesN/A_ GC/MS TuningN/A_ Internal Standard PerformanceX BlanksN/A_ Surrogate RecoveriesN/A_ Matrix Spike/Matrix Spike Duplicate	X Laboratory Control SpikesX Field DuplicatesX CalibrationsX Compound IdentificationsX Compound QuantitationX Quantitation Limits
Overall Comments:_Methane_by_ASTM_method_D-19	46_(modified)
Definition of Qualifiers: J- Estimated results U- Compound not detected R- Rejected data UJ- Estimated nondetect	
Reviewer: (afaul Mauf Date:01/07/2015	··-

DATA COMPLETENESS

MISSING INFORMATION	DATE LAB. CONTACTED	DATE RECEIVED
		* * * * * * * * * * * * * * * * * * * *
	*	

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		The state of the s
		

All criteria were metX
Criteria were not met
and/or see below

HOLDING TIMES

The objective of this parameter is to ascertain the validity of the results based on the holding time of the sample from time of collection to the time of analysis.

Complete table for all samples and note the analysis and/or preservation not within criteria

SAMPLE ID	DATE SAMPLED	DATE ANALYZED	рН	ACTION
	Ali samples analyzed w	ithin the recommended	i method	holding time

Criteria

Aqueous samples – 14 days from sample collection for preserved samples (pH \leq 2, 4°C), no air bubbles.

Aqueous samples – 7 days from sample collection for unpreserved samples, 4°C, no air bubbles. Soil samples- 7 days from sample collection.

Cooler temperature (Criteria: 4 + 2 °C): N/A – summa canisters

Actions

If the VOCs vial(s) have air bubbles, estimate positive results (J) and reject nondetects (R).

If the % solids of soil samples is 10-50%, estimates positive results (J) and nondetects (UJ)

If the % solid of soil samples is < 10%, estimate positive results (J) and reject nondetects (R).

If holding times are exceeded but < 14 days beyond criteria, estimate positive results (J) and nondetects (UJ).

If holding times are exceeded but < 28 days beyond criteria, estimate positive results (J) and reject nondetects (R).

If holding times are grossly exceeded (> 28 days beyond criteria), reject all results (R).

If samples were not iced or if the ice were melted (> 10°C), estimate positive results (J) and nondetects (UJ).

		Crite	All criteria were metN/A eria were not met see below
GC/MS TUNING			
The assessment standard tuning C		determine if the sample instru	mentation is within the
N/A_ The BFB	performance results were	reviewed and found to be within	the specified criteria.
N/A_ BFB tunir	ng was performed for every	24 hours of sample analysis.	
If no, use profest qualified or rejecte		ine whether the associated data	a should be accepted,
List	the	samples	affected:
If mass calibration	n is in error, all associated	data are rejected.	

Note: Samples analyzed using GC with either TCD or FID detection.

4

All criteria were metX
Criteria were not met
and/or see below

CALIBRATION VERIFICATION

Compliance requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing and maintaining acceptable quantitative data.

Date of initial calibration:	05/08/14
Dates of continuing calibra	tion:_12/15/14
Instrument ID numbers:	GC-9
Matrix/Level:	Air/low

DATE	LAB ID#	FILE	CRITERIA OUT RFs, %RSD, %D, r	COMPOUND	SAMPLES AFFECTED
			rations meet method sprequirements.	pecific requirements. Initia	calibration retention
unica meci	inculou	эреспи	requirements.		

Criteria

All RFs must be > 0.05 regardless of method requirements for SPCC.

All %RSD must be ≤ 15 % regardless of method requirements for CCC.

All %Ds must be < 30% regardless of method requirements for CCC.

Method TO-15 does not specify criterion for the curve correlation coefficient (r). A limit for r of \geq 0.995 has therefore been utilized as professional judgment.

Actions

If any compound has an initial RF or a continuing RF of < 0.05, estimate positive results (J) and reject nondetects (R), regardless of method requirements.

If any compound has a %RSD > 15%, estimate positive results (J) and use professional judgment to qualify nondetects.

If any compound has a %RSD > 90%, estimate positive results (J) and reject nondetects (R).

If any compound has a % D > 30%, estimate positive results (J) and reject nondetects (R).

If any compound has a % D > 30%, estimate positive results (J) and nondetects (UJ).

If any compound has a % D > 90%, estimate positive results (J) and reject nondetects (R).

If any compound has r > 0.995, estimate positive results and nondetects.

A separate worksheet should be filled for each initial curve

All criteria were metX
Criteria were not met
and/or see below

V A. BLANK ANALYSIS RESULTS (Sections 1 & 2)

The assessment of the blank analysis results is to determine the existence and magnitude of contamination problems. The criteria for evaluation of blanks apply only to blanks associated with the samples, including trip, equipment, and laboratory blanks. If problems with any blanks exist, all data associated with the case must be carefully evaluated to determine whether or not there is an inherent variability in the data for the case, or if the problem is an isolated occurrence not affecting other data.

List the contamination in the blanks below. High and low levels blanks must be treated separately.

Laboratory blanks

DATE ANALYZED	LAB ID	LEVEL/ MATRIX	COMPOUND	CONCENTRATIO UNITS
All_metho	 d_blank_meeth	 _method_speci	ific_criteria	
Summa_c			ation_criteria	
Field/Equipmer				
DATE ANALYZED	LAB ID	LEVEL! Matrix	COMPOUND	CONCENTRATION UNITS
No_field/trip/eq	uipment_blanks	_analyzed_with	n_this_data_package	
			- · · · · · · · · · · · · · · · · · · ·	

Alf criteria were metX	
Criteria were not met	
and/or see below	

VB. BLANK ANALYSIS RESULTS (Section 3)

Blank Actions

Action Levels (ALs) should be based upon the highest concentration of contaminant determined in any blank. Do not qualify any blank with another blank. The ALs for samples which have been diluted should be corrected for the sample dilution factor and/or % moisture, where applicable. No positive sample results should be reported unless the concentration of the compound in the samples exceeds the ALs:

ALs = 10x the amount of common contaminants (methylene chloride, acetone, 2-butanone, and toluene)

ALs = 5x for any other compounds

Specific actions are as follows:

If the concentration is < sample quantitation limit (SQL) and \le AL, report the compound as not detected (U) at the SQL.

If the concentration is \geq SQL but \leq AL, report the compound as not detected (U) at the reported concentration.

If the concentration is \geq SQL and > AL, report the concentration unqualified.

Notes:

High and low level blanks must be treated separately Compounds qualified "U" for blank contamination are still considered "hits" when qualifying for calibration criteria.

CONTAMINATION SOURCE/LEVEL	COMPOUND	CONC/UNITS	AL/UNITS	SQL	AFFECTED SAMPLES
					9e
				1	
			,		
Again.					

All criteria were metN/A
Criteria were not met
and/or see below

ACTION

SURROGATE SPIKE RECOVERIES

Laboratory performance of individual samples is established by evaluation of surrogate spike recoveries. All samples are spiked with surrogate compounds prior to sample analysis. The accuracy of the analysis is measured by the surrogate percent recovery. Since the effects of the sample matrix are frequently outside the control of the laboratory and may present relatively unique problems, the validation of data is frequently subjective and demands analytical experience and professional judgment.

SURROGATE COMPOUND

List the percent recoveries (%Rs) which do not meet the criteria for surrogate recovery. Matrix: solid/aqueous

_Surrogate_standaro	ds_not_requir	red_by_the_met	hod		

QC Limits* (Air) LL to UL	to	to ·	to	to	

- * QC limits are laboratory in-house performance criteria, LL = lower limit, UL = upper limit.
- * If QC limits are not available, use limits of 80 120 % for aqueous and 70 130 % for solid samples.

Actions:

SAMPLE ID

QUALITY	%R < 10%	%R = 10% - LL	%R > UL
Positive results	J	J	J
Nondetects results	R	UJ	Accept

Surrogate action should be applied:

If one or more surrogate in the VOC fraction is out of specification, but has a recovery of > 10%.

If any one surrogate in a fraction shows < 10 % recovery.

All criteria were met
Criteria were not met
and/or see belowN/A

VII. A MATRIX SPIKE/MATRIX SPIKE DUPLICATE (MS/MSD)

This data is generated to determine long term precision and accuracy in the analytical method for various matrices. This data alone cannot be used to evaluate the precision and accuracy of individual samples. If any % R in the MS or MSD falls outside the designated range, the reviewer should determine if there are matrix effects, i.e. LCS data are within the QC limits but MS/MSD data are outside QC limit.

1. MS/MSD Recoveries and Precision Criteria

The laboratory should use one MS and a duplicate analysis of an unspiked field sample if target analytes are expected in the sample. If target analytes are not expected, MS/MSD should be analyzed.

List the %Rs, RPD of the compounds which do not meet the criteria.

Sample ID:		Matrix/Level:				
MS OR MSD	COMPOUND	% R	RPD	QC LIMITS	ACTION	
	_not_required_as_part	_of_ASTM	1-method	_D-1946;_blan	k_spike_used_to_assess	

- * QC limits are laboratory in-house performance criteria, LL = lower limit, UL = upper limit.
- * If QC limits are not available, use limits of 70 130 %.

Actions:

QUALITY	%R < LL	%R > UL
Positive results	J	J
Nondetects results	R	Accept

MS/MSD criteria apply only to the unspiked sample, its dilutions, and the associated MS/MSD samples:

If the % R for the affected compounds were < LL (or 70 %), qualify positive results (J) and nondetects (UJ).

If the % R for the affected compounds were > UL (or 130 %), only qualify positive results (J).

If 25 % or more of all MS/MSD %R were < LL (or 70 %) or if two or more MS/MSD %Rs were < 10%, qualify all positive results (J) and reject nondetects (R).

A separate worksheet should be used for each MS/MSD pair.

All criteria were met
Criteria were not met
and/or see belowN/A

VII. B MATRIX SPIKE/MATRIX SPIKE DUPLICATE

MS/MSD - Unspiked Compounds

It should be noted that Method TO-15 does not specify a MS/MSD criteria for the unspiked compounds in the sample. A %RSD of < 50% has therefore been utilized as professional judgment.

If all target analytes were spiked in the MS/MSD, this review element is not applicable.

List the %RSD of the compounds which do not meet the criteria.

Sample ID:			Matrix/Level/Unit			
COMPOUND	SAMPLE CONC.	MS CONC.	MSD CONC.	% RSD	ACTION	
		V 7.00	<u> </u>			
V						
		· · · · · · · · · · · · · · · · · · ·				
Alex.						

Actions:

^{*} If the % RSD > 50, qualify the positive result in the unspiked samples as estimated (J).

^{*} If the % RSD is not calculated (NC) due to nondetected value, use professional judgment to qualify the data.

All criteria were metX
Criteria were not met
and/or see below

VIII. LABORATORY CONTROL SAMPLE (LCS) ANALYSIS

This data is generated to determine accuracy of the analytical method for various matrices.

1. LCS Recoveries Criteria

Where LCS spiked with the same analyte at the same concentrations as the MS/MSD? Yes or No. If no make note in data review memo.

List the %R of compounds which do not meet the criteria

	LCS ID	COMPOUND	% R	QC LIMIT
LCS/I	_CSD_(Blank_spik	ce)_analyzed_in_this_data_	_package;_recoveries_	and_RPD
within	_laboratory_contro	ol_limits		
		V		

- * QC limits are laboratory in-house performance criteria, LL = lower limit, UL = upper limit.
- * If QC limits are not available, use limits of 70 130 %.

Actions:

QUALITY	%R < LL	%R > UL
Positive results	J	J
Nondetects results	R	Accept

All analytes in the associated sample results are qualified for the following criteria.

If 25 % of the LCS recoveries were < LL (or 70 %), qualify all positive results (j) and reject nondetects (R).

If two or more LCS were below 10 %, qualify all positive results as (J) and reject nondetects (R).

2. Frequency Criteria:

Where LCS analyzed at the required frequency and for each matrix? <u>Yes</u> or No. If no, the data may be affected. Use professional judgment to determine the severity of the effect and qualify data accordingly. Discuss any actions below and list the samples affected.

			All criteria were metX Criteria were not met and/or see below
IX.	LABORATORY	DUPLICATE PRECISION	
	Sample IDs:	1412151B-02A/1412151B-03A	Matrix:_Air

Field duplicates samples may be taken and analyzed as an indication of overall precision. These analyses measure both field and lab precision; therefore, the results may have more variability than laboratory duplicates which only laboratory performance. It is also expected that soil duplicate results will have a greater variance than water matrices due to difficulties associated with collecting identical field duplicate samples.

The project QAPP should be reviewed for project-specific information. Suggested criteria: RPD <u>+</u> 25% for air samples. If both samples and duplicate are <5 SQL, the RPD criteria is doubled.

COMPOUND	SQL	SAMPLE CONC.	DUPLICATE CONC.	RPD	ACTION
	RPD v	vithin the me	thod performand	e criter	ia.

Actions:

Qualify as estimated positive results (J) and nondetects (UJ) for the compound that exceeded the above criteria. For organics, only the sample and duplicate will be qualified.

If an RPD cannot be calculated because one or both of the sample results is not detected, the following actions apply:

If one sample result is not detected and the other is greater than 5x the SQL qualify (J/UJ).

If one sample value is not detected and the other is greater than 5x the SQL and the SQLs for the sample and duplicate are significantly different, use professional judgment to determine if qualification is appropriate.

If one sample value is not detected and the other is less than 5x, use professional judgment to determine if qualification is appropriate.

If both sample and duplicate results are not detected, no action is needed.

All criteria were metN/A
Criteria were not met
and/or see below

X. INTERNAL STANDARD PERFORMANCE

The assessment of the internal standard (IS) parameter is used to assist the data reviewer in determining the condition of the analytical instrumentation.

List the internal standard area of samples which do not meet the criteria.

- * Area of +40% or -40% of the IS area in the associated calibration standard.
- * Retention time (RT) within \pm 0.06 seconds of the IS area in the associated calibration standard.

DATE	SAMPLE ID	IS OUT	IS AREA	ACCEPTABLE AC RANGE	TION
_Internal_sta _method	andard_not_require	d_by_the_metho	odSamples_qua	nntified_by_external_sta	ndard
Actions:					

1. IS actions should be applied to the compound quantitated with the out-of-control ISs

QUALITY	IS AREA < -40%	IS AREA > + 40%
Positive results	J	J
Nondetected results	R	ACCEPT

2. If a IS retention time varies more than 0.330 seconds, the chromatographic profile for that sample must be examined to determine if any false positive or negative exists. For shifts of a large magnitude, the reviewer may consider partial or total rejection of the data for the sample fraction.

ΑII	criteria	were	met	х
	4.110114		,,,,	

Criteria were not met	
and/or see below	

XII. SAMPLE QUANTITATION

The sample quantitation evaluation is to verify laboratory quantitation results. In the space below, please show a minimum of one sample calculation:

1412151B-01A

Methane

RF = 159806712

[] = (196558)/(159806712)

= 0.0012 % OK

All criteria were metX
Criteria were not met
and/or see below

- XII. QUANTITATION LIMITS
- A. Dilution performed

SAMPLE ID	DILUTION FACTOR	REASONS FOR DILUTION			
All samples diluted by a factor of 2					
•••					
	•				
64.00 mg	<u> </u>				

1000			
B.	Percent S	Solids	
	List samp	les which have ≤ 50°	% solids
	V. 1035		
Actions		olids of a soil sample	e is 10-50%, estimate positive results (J) and nondetects (UJ)
	If the % s (R)	olids of a soil sample	e is < 10%, estimate positive results (J) and reject nondetects